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OXYGEN CONCENTRATION, TEMPERATURE, AND VISCOSITY DETERMINATIONS

Polarographic Technic

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FOREWORD

This report was prepared in the Radiobiology Branch under task No. 571003. It was submitted for publication on 26 October 1965. The work was accomplished between June and October 1965.

This report has been reviewed and is approved.

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ABSTRACT

A technic was developed for determining the effects of ionizing radiation on molecular oxygen concentration in an aqueous solution. The polarographic principle was used for determining oxygen concentration, temperature, and viscosity of solution, and oxygen concentration in gas. The polarographic system has the following principal characteristics: (1) parameters can be remotely measured, permitting the system to be used in environments hostile to observers (i.e., radiation); (2) temperature measurement can be made within 1° C.; (3) changes in temperature are recorded instantly; (4) oxygen concentration can be measured with a resolution well within 1 mm. Hg; and (5) oxygen changes are recorded instantly.

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I. INTRODUCTION

The polarographic method of determining oxygen concentration in gaseous and solution environments has been used many times (1). Use of polarography to measure oxygen concentration has been limited in the past to oxygen measurement. The influence of temperature, viscosity, and fluid movement on the diffusion coefficient of oxygen has been investigated (2-5), and a knowledge of these variables must be ascertained at the time the oxygen measurement is made, if the oxygen measurement is to be accurate. If all but one of these variables (e.g., viscosity) were maintained constant, the system would then measure the coefficient of viscosity of the solution. Likewise, the principle could be applied to determine quantitative values of such other variables as temperature, dynamics of fluid movement, and oxygen concentration. This communication describes the use of polarography to measure temperature, oxygen concentration, and viscosity.

II. METHODS

Apparatus

The apparatus that is used to measure the parameters listed in the introduction is illustrated in figure 1. Both cell *A* and cell *B* can be used interchangeably as test and control cells. *C* represents a solenoid valve that can permit the control of the system remotely, and *D* indicates a tank of gas that can be used to change the concentration of oxygen in the fluid medium in either cell. Only two cells and one cylinder are shown in this figure, but the system has the capability of being extended to utilize simultaneously many gas cylinders and many cells.

A close-up of the cell, in which may be seen the protrusion of platinum and silver electrodes into a saline solution, is shown in figure 2. The cover over cell *A* provides a means for controlling the gaseous environment over the solution. The leads extending from the electrodes are coupled to a polarograph that can be located far enough from the test cell to permit this system to be used in environments hostile to the observer (i.e., radiation).

Instrumentation

The polarographic circuit (fig. 3) used in the system involves the following principle: oxygen is reduced at the cathode (platinum electrode), and electron flow resulting from this reduction is measured as a voltage drop across resistor *A*, *B*, or *C*. The basic equation for this principle is:

$$O_2 + 2e + 2H^+ \rightarrow H_2O_2 + 2e + 2H^+ \rightarrow 2H_2O$$

The measurements are made in a dilute solution of an electrolyte (e.g., 0.9 *N* NaCl). The external current control shown in the circuit was not used in this study; however, it provides the capability of applying to the cathode an a.c. voltage rather than a d.c. voltage. In certain experiments, it is advantageous to use an a.c. voltage owing to the effect of contamination upon the electrode by the medium in which the electrode is inserted (1). The circuit on the positive side of the output is used primarily to suppress the voltage differences that are created in the cell by the combination of platinum, silver, and sodium

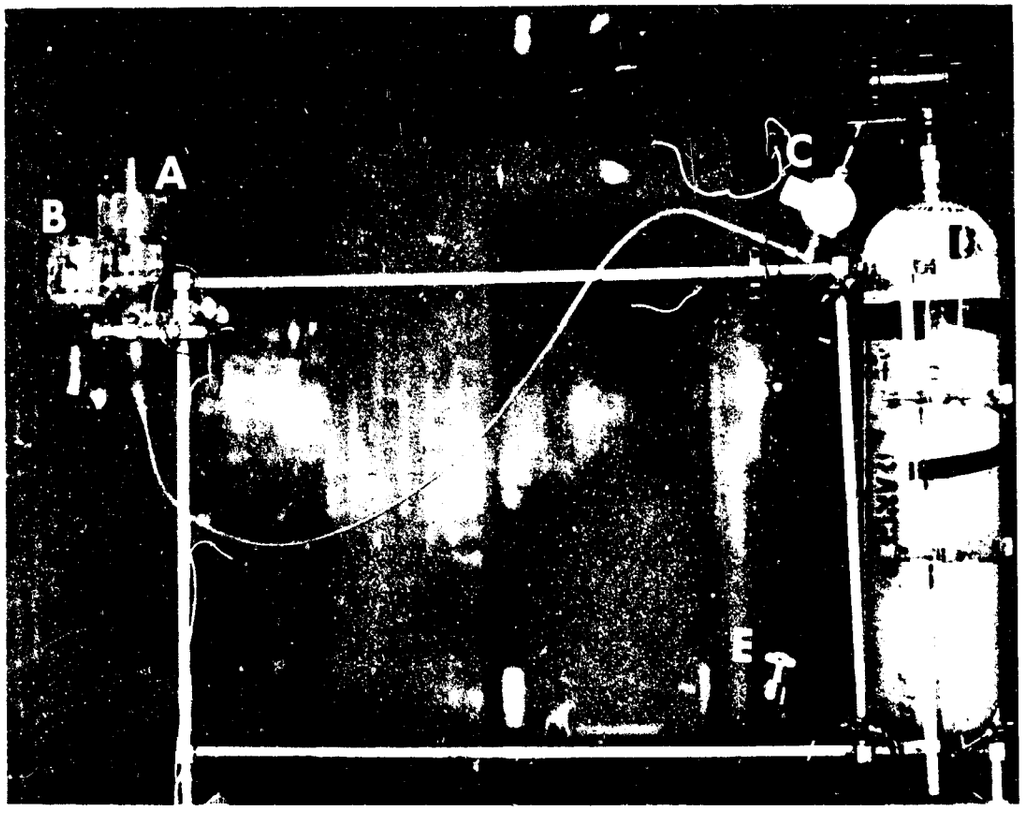

FIGURE 1

Cell A *with cover to control atmosphere over the solution. Cell* B *can be used with or without the cover. Solenoid valve* (C) *can be remotely operated. Tank containing nitrogen* (D)*; bubbling nitrogen through the glass filter in cell* A *removes the oxygen. Extension cable connected to* E *permits remote control of solenoid valve.*

chloride, thereby permitting at the output for further amplification only oxygen-reduction current. This arrangement makes possible a resolution of oxygen concentration within 1 mm. Hg.

In this system, the applied voltage on the platinum electrode is 1 v., permitting the system to function on the plateau of the current-voltage curve (fig. 4).

A thermistor is used to measure the temperature of the saline solution in the test cell (see block diagram, fig. 5). A glass filter permits the passage of gas into the cell and prohibits the leakage of saline solution from the cell. The reactor core is shown to indicate a possible use of all the remote features of the system. The low level of the voltage drop produced by the oxygen-reduction current across the resistors (indicated in fig. 3) makes it necessary to use d.c. amplifiers to gain an acceptable degree of resolution. With this system, changes of 1 mm. Hg can be detected. Also, the system permits easy determination of temperature changes within 1° C.

III. RESULTS AND DISCUSSION

Oxygen concentration

It is well known that polarographic systems can measure the concentration of oxygen in fluid and tissue mediums (6, 7, 8). The use of nitrogen to reduce the oxygen concentration in a saline solution and the use of air to return the solution to normal oxygen concentration are illustrated in figure 6. The oxygen-reduction current is increased with the onset of the nitrogen bubbles that are forced through the

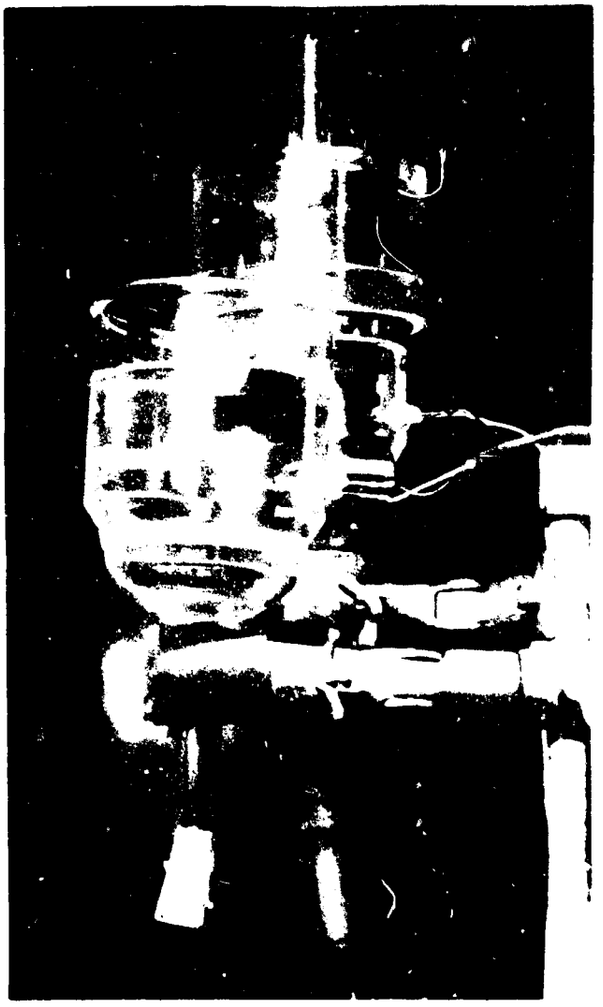

FIGURE 2

Test and control cells indicating the protrusion of platinum-wire and silver-wire electrodes into a normal solution of sodium chloride. Glass filter is located at the bottom of the cell (white ring). The cover A *over control cell* C *permits the environmental gas to remain constant.* B *is close-up of the test cell.*

glass filter. This increase in the oxygen-reduction current is typical of all bare oxygen-sensitive electrodes and is due to a phenomenon known as "stirring potentials" (constant changing of the oxygen-diffusion gradient). Also, it may be noted in figure 6 that when oxygen was returned to the solution, the oxygen concentration exceeded its original value. This, too, was due to the effect of stirring potentials.

To measure the unknown oxygen concentration in a solution or in gas, the following steps are required:

1. Calibrate the polarographic system by measuring the oxygen-reduction current flow in a saline solution in air. This current flow represents 150 mm. Hg (20% oxygen in air, or 20% of 750 mm. Hg). Bubble nitrogen through the glass filter, removing all oxygen in the saline solution. If proper use is made of the circuit on the positive-output side of the schematic shown in figure 3, no current flow will be detected after nitrogen is bubbled through the solution for several minutes. The relationship is linear between oxygen current flow and oxygen concentration.

2. Force the gas containing the unknown oxygen concentration through the saline solution equilibrated to oxygen concentration in air, and use the calibration curve (plotted as described above) to determine the current flow resulting from the gas mixture in the solution, thus ascertaining the oxygen concentration in the gas mixture.

3. Replace the saline solution with a solution containing the unknown oxygen concentration and measure the oxygen-reduction current flow. With this value and the calibration curve, determine the oxygen concentration in the solution. The viscosity and temperature of the saline solution used to calibrate the system and of the solution containing the unknown oxygen concentration must be the same.

Temperature

The polarographic system as a temperature-measuring device was compared with a thermistor (fig. 7A). The temperature change was produced in a medium in which these curves were made simultaneously in each system. The thermistor lagged the polarographic system by approximately 0.5 sec.; therefore, when one is interested in measuring instantaneous temperature changes, the polarographic method for making these measurements is far superior to a system in which a thermistor is used. The difference in the sensitivity of the two systems is apparent. The actual change in temperature was approximately 10° C.; however, it is obvious from

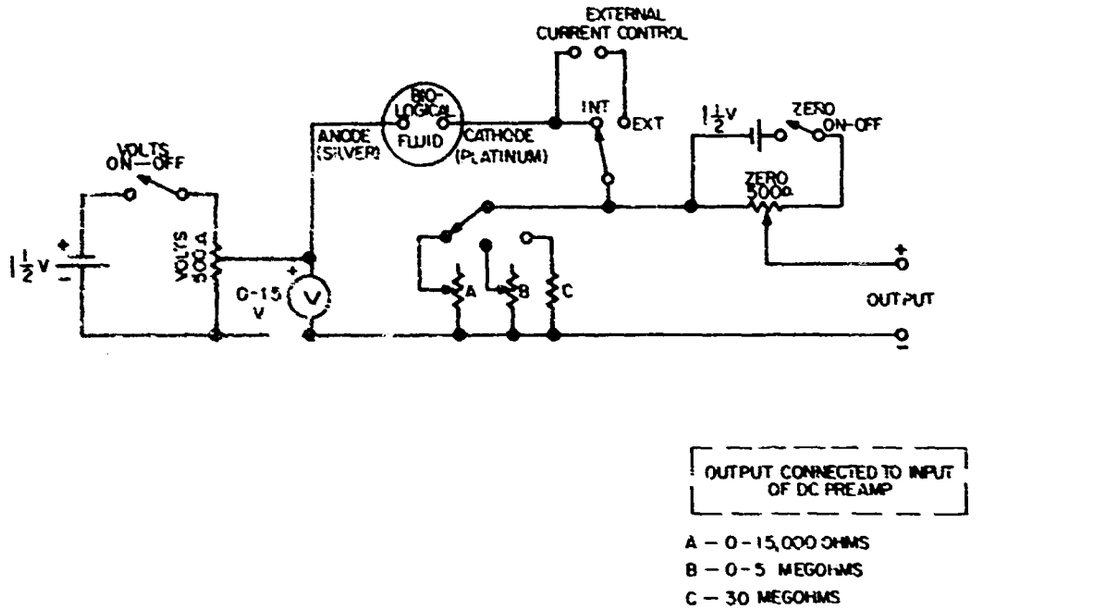

FIGURE 3

Polarographic circuit. The example "biologic fluid" represents any medium containing oxygen in molecular form. Zero suppression, positive side of output, permits greater sensitivity and provides determination within 1 mm. Hg or 1° C. change. Selection of resistor **A, B, or C** *changes the output sensitivity.*

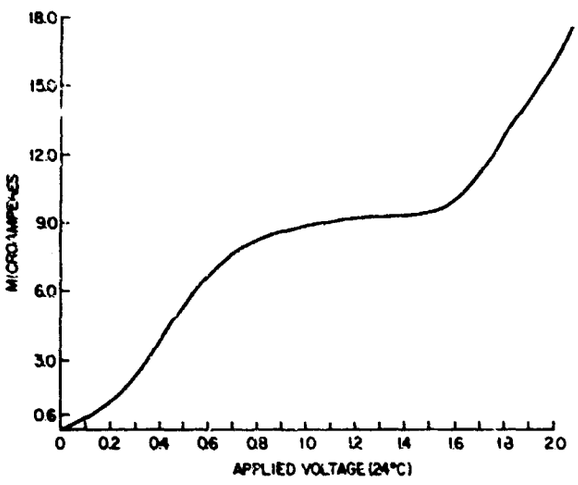

FIGURE 4

Typical current-voltage curve.

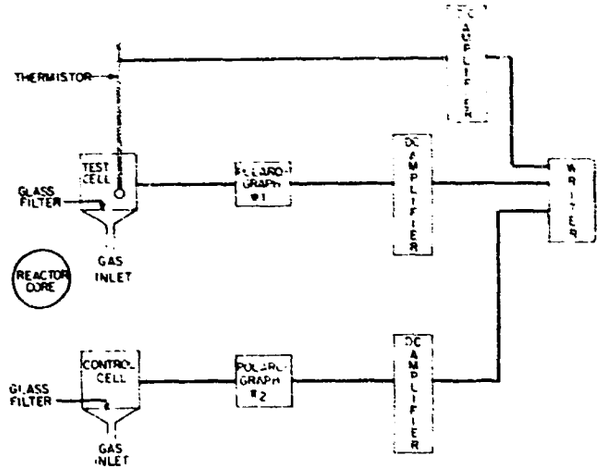

FIGURE 5

Block diagram indicating a reactor core, to demonstrate a use requiring the remote features of the system.

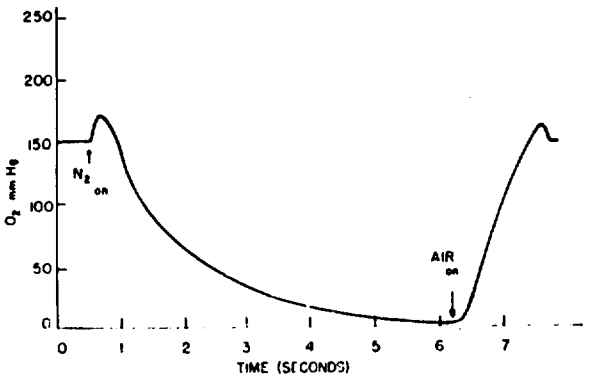

FIGURE 6

System's response curve to oxygen reduction.

the magnitude of the change when the polarographic method is used that a resolution can be made much less than 1° C.

An experiment was performed to indicate the necessity for oxygen when the system is used as a temperature indicator (fig. 7B). Two things are apparent: (1) the oxygen must be present, and (2) the temperature-sensitivity of the system (the degree of resolution with respect to temperature change) depends on the magnitude of the oxygen concentration; the higher the oxygen concentration, the more sensitive the system is to temperature changes. When the oxygen concentration reaches zero, the system is no longer sensitive to temperature changes. As shown in figure 7B, at approximately 6.5 sec. the temperature in the solution was changed and the thermistor (shown by a dotted line) indicated a change of approximately 10° C. During this period, however, bubbling nitrogen through the solution had removed the oxygen from the system at 1.0 sec., and no change due to temperature variation in the solution was noted in the polarographic oxygen curve.

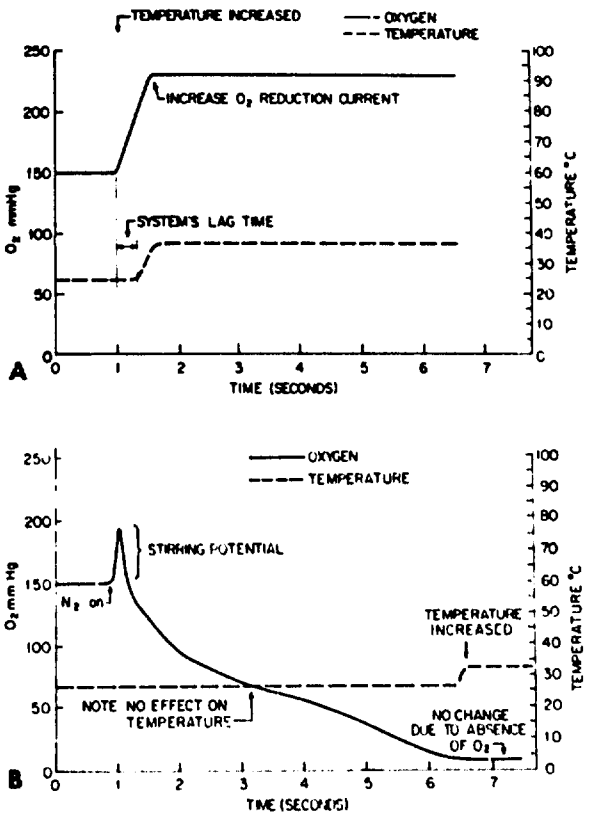

FIGURE 7

Polarographic system used as a temperature indicator. A, *Note instantaneous reaction of the polarographic system to temperature changes. Reaction time lag between the two systems is approximately 0.5 sec.*

B, *Note the necessity for the presence of oxygen to measure temperature change. Thermistor registered temperature change (dotted line), but change was absent in the polarographic temperature curve (solid line).*

Viscosity

The polarographic system was used to measure the viscosity of a solution (fig. 8). Saline was added to the control cell each time a mixture of agar was added to the test cell. No change in the voltage of the polarographic system was noted in the control cell when saline was added; however, each addition of agar solution to the test cell decreased the voltage output of the test cell. It is important to realize at this point that, even though the oxygen-diffusion current had been reduced, the oxygen concentration was maintained at 150 mm. Hg. Each addition of agar essentially changed the oxygen-diffusion coefficient, producing a lower voltage for the same oxygen concentration.

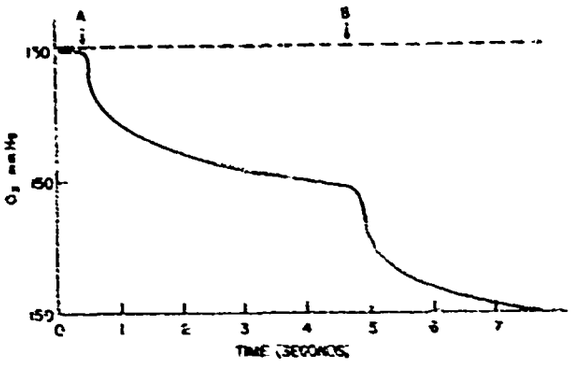

FIGURE 8

Polarographic system used to measure viscosity. A and B, Saline solution added to control cell containing saline (dotted line) and agar added to test cell containing agar solution (solid line).

When this method is used to measure viscosity, it is imperative that the temperature and the oxygen concentration be unchanged during the measurement. Once a solution with known viscosity has been added and the system has been calibrated, the test cell will be ready for determination of the unknown viscosity. Additions of a solution in which the viscosity is unknown into a test cell should register a voltage output of the system which, when matched with the calibration curve, should give a fair approximation of the viscosity of the new solution.

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